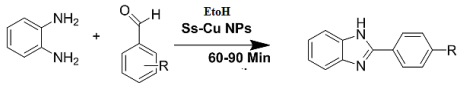
Silica Supported Copper Nano Particles Design from Metal Waste Apply Formation of Various Condensation Reaction

Umesh Nitone[1], Jayaveersinh Mahida[2*], Seema Agrawal[1], Ravi Patel[2]

[1]Department of chemistry, Dr. A P J Abdul Kalam University, Indore (MP), India.
[2]Applied chemistry, Shree P. M. Patel Institute of P.G. Studies & Research in Science (Sardar Patel university), Sardar patel University, Anand ,India
*Corresponding Author: rsjkmappchem1417@gmail.com

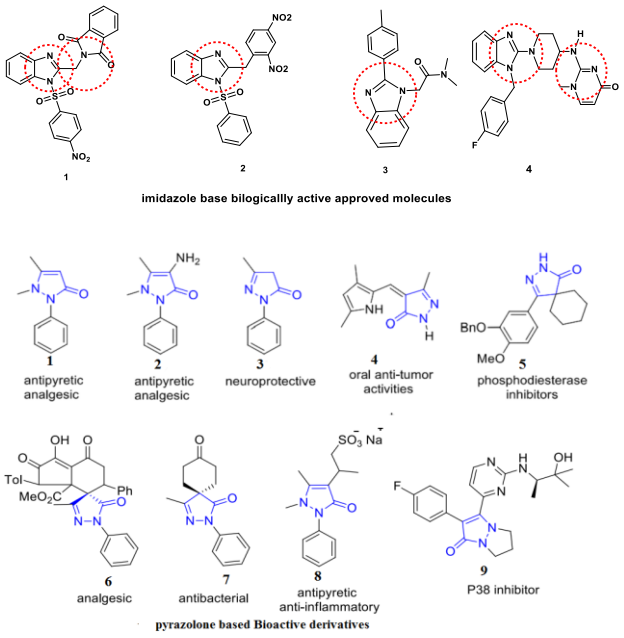
Abstract. In recent years organic chemist focused on green chemistry for organic synthesis transformations because the silica supported catalyst is more stable, efficient and easy to recover and reusable after reaction. The silica supported copper catalyst is design from E-Waste, It used for formation of benzimidazoles from diamines and aldehydes.it is very deep work done by various catalyst, we noted in our research in unique method develop for synthesis of benzimidazoles made by condensation diamine and aldehydes via silica supported copper nano particles under EtOH as reaction media . The reactions done at 60° C temperature for 80 minute by 1:1.2 ratio mixture of diamine and aldehydes in the presence of nano catalyst to give the desired products with high purity..



Index Terms: Schame: 01 **; Scheme 1.** Synthesis of benzimidazole derivatives;
Waste management, green catalyst, high reusability, Biological evaluation

I. INTRODUCTION

Currently metal waste, plastic waste and e-waste is highest global environment issue ,House hold electronic gadgets PCB is made by copper wire, the gadgets are out of service its produced electronic waste, our team is collected this type house hold e-waste and design it's chemical process for formation of nano particles, it nano particles apply as a catalyst for formation of various know biologically active heterocyclic derivatives like benzimidazole, s via solvent free or aqueous media condition . Checked efficiency and reusability of catalyst. the all derivatives are based on the heterocyclic chemistry and highly applicable as lead molecules of biological activities of benzimidazole like anti HBV (1) [5], antitubercular (2) [6], non-seadating antihistamine(3) [7], (GABAA agonists [8], anti-viral (3) [9], antiinflammatory and analgesic [10], The drug discovery application of pyrazolone includes antipyretic activity (1,2,3,4) and analgesic activity (1,2,5,7,8,9),12,13,14,15 antibacterial ,16 anti-inflammatory activity antitumor activity (7,8,9),17,18 aneuroprotective and cardiovascular agent.



II. MATERIALS AND METHODS

A mixture of o-phenylenedianmine (5 mmol) and sub.aldehyde (5.5 mmol) was stirred for 15 min than add novel designed Silica supported copper nano particles as catalyst at 28°C and use 5 ml Ethanol as a solvent at 60°C refluxed 80 to 90 minute and check TLC in Hexane: Ethyl acetate: Acetic acid(70:20:10) and reaction if complies, clear reaction mass filter by filter

© 2022 Technoarete Publishers
Umesh Nitone, Jayaveersinh Mahida, Seema Agrawal, Ravi Patel – "Silica Supported Copper Nano Particles Design from Metal Waste Apply Formation of Various Condensation Reaction", Pg no: 44 – 47. 2022
doi.org/10.36647/978-93-92106-02-6.9

paper and remove catalyst than solvent remove from product and repurify in Ethanol and check its Melting point and TLC, stucture determination by IR by FTIR,^{1}HNMR,^{13}CNMR and mass spectroscopy.

III. RESULTS AND DISCUSSION

We initiated our observation by using diamine (1.0 equiv.) and aromatic aldehyde (1.02 equiv.) in presence of silica supported copper nano particles (1-3%) in ethanol solvent at 55-65°C under reflux condition. the reaction goes out forward direction 80-85 % of conversion and 10% KSM and 5% some unknown impurities , were isolated within 2 h. the amount increased of catalyst to 10 mol% notable significantly with 86 % of **product** and14 % of **KSM** (Table 1, entry 2). Furthermore amount of catalyst does not affect on yield of the reaction (Table 1, entry 3).other parameters like time and Temperatures does not affect on yield .**4b** and **5** decreased considerable. Increasing temperature from 60 °C does not possible in Ethanol solvent.

Table 1. Optimization of the reaction conditions and EtOH used as a solvent [1].

Entry	Catalyst(Mole%)	Temp(°C)	Time	Yield	Purity	KSM	impurity
1	3	60	240	65%	72	08	20
2	5	60	80	87%	92	05	03
3	7	60	80	85%	89	05	06
4	10	60	80	81%	85	04	08
5	12	60	80	83%	87	05	08
6	15	60	80	84%	88	04	09
7	20	60	80	82%	85	12	03
8	CuCl3	60	240	NR	--	--	--
9	Cu(OAc)	60	240	NR	--	--	--
10	Cu(OTf)2	60	240	NR	--	--	--
11	CuO	60	240	55%	78	16	06

All experiment purity based on First crop of the reaction and all analysis done by TLC w/w concretions method. NR: No reactions

After the optimization of reaction condition and catalyst mole 5% and 4 volume solvent EtoH.

Table 2 optimization of reaction for different derivatives

Entry	Aldehyde	Time	Yield(%)	MP°C	MP°C(Reported)
1	Benzaldehyde	**60**	**84**	**292**	**292-294**
2	Isobutyraldehyde	**80**	**89**	**236**	**235-236**
3	4-Chlorobenzaldehyde	**70**	**84**	**266**	**265-267**
4	trans-Cinnamaldehyde	**70**	**89**	**202**	**201-203**
5	Hexanal	**90**	**88**	**163**	**164-165**

In recent years organic chemist focused on green chemistry for organic synthesis [29] because the silica supported catalyst is more stable, efficient and easy to recover and reusable after reaction. The silica supported copper catalyst is design from E-Waste, It formation of benzimidazoles from diamines and aldehydes. it is very known work for check the catalyst activity [30] , we noted in our research in novel method develop for formation of benzimidazoles by the condensation of diamine and aldehydes react via catalytic amount of silica supported copper nano particles under EtOH as reaction media . The reactions done at 60° C temperature for 60 to 90 min by taking a 1:1.2 mol ratio mixture of diamine and aldehydes in the presence of 5 mol% catalyst to give the desired products in and notable (Scheme 1). As shown in Table 2, aromatic, aliphatic, and unsaturated aldehydes use as substituent for reaction react without any difficulty and gave approx same yield and near similar time, difference in rates to give the corresponding 2-substituted benzimidazoles superb yield..

Supportive Spectroscopic data:

Entry 01:

pale yellow solid, FT-IR (KBr) 3450, 3042, 2910, 2100, 1630, 1580, 1470, 1423, 1373, 1202, 1118, 980, 821cm-1 ^{1}H NMR (400 MHz, DMSO-d6) : 8.17 (2H), 7.70-7.32 (5H), 7.10-7.00 (1H), 2.47 (3H); ^{13}C NMR (100 MHz, DMSO-d6) :149.9, 142.2, 141.0, 132.3, 134.0, 132.1, 130.7, 125.7, 122.4, 117.2, 110.1, 20.4;. MS (ESI) m/z: [M+H]+ calcd :209.10, found 211.

Entry 02:

Greenish solid, FT-IR (KBr):1630, 1580, 1437, 1434, 1320,cm-1 ^{1}H NMR (400 MHz, DMSO-d6) : 7.70-7.12 (2H), 7.20-7.00 (2H), 3.24(1H)1.27 (6H); ^{13}C NMR (100 MHz, DMSO-d6) : 141.10, 138.2, 128.7,115.2,29.2, 20.3;. MS (ESI) m/z: [M+H]+ calcd :161.10, found 160.2.

Entry 03:

pale yellow solid; FT-IR (KBr) 3441, 2997, 2949,2110, 1626, 1585, 1487, 1422, 1310, 1270, 1220, 1108, 1010cm-1^{1}H NMR (400MHz, DMSO-d6) :12.90(1H), 8.19 (2H), 7.70 (1H), 7.64 (2H), 7.50 (1H), 7.25-7.20

(2H); ^{13}C NMR (100 MHz, DMSO-d6) :149.1,142.9, 135.1, 131.4, 130.2, 129.7, 128.1, 121.7, 121.2, 118.1, 111.4, MS (ESI) m/z:[M+H]+ calcd 229.05, found 230.23.

REFERENCES

1. For some recent reviews on directed C-H functionalization reactions, see: (a) Colby, D. A.; Bergman, R.G.; Ellman, J. A. *Chem. Rev*. **2010**, *110*, 624. (b) Wencel-Delord, J.; Droge, T.; Liu, F.; Glorius, F. *Chem.Soc. Rev*. **2011**, *40*, 4740. (c) Wendlandt, A. E.; Suess, A. M.; Stahl, S. S. *Angew. Chem., Int. Ed.* **2011**,*50*, 11062. (d) Engle, K. M.; Mei, T.-S.; Wasa, M.; Yu, J.-Q. *Acc. Chem. Res.* **2012**, *45*, 788. (e)
2. Hickman, A. J.; Sanford, M. S. *Nature Rev.* **2012**, *484*, 178. (f) Wencel-Delord, J.; Glorius, F. *Nature Chem.* **2013**, *5*, 369.
3. Afaf, H.E., Fahmy, H.H., Abdelwal, S.H., 2000. Molecules 5, 1429. Amari, M., Fodili, M., Nedjar-kolli, B., 2002. Reactivity studies on 4-aminopyrones: Access to benzimidazole and benzimidazolone derivatives. J. Heterocycl. Chem. 39, 811
4. Ansari, K.F., Lal, C., 2009a. Synthesis and evaluation of some newbenzimidazole derivatives as potential antimicrobial agents. Eur. J.Med. Chem. 44, 2294.Ansari, K.F., Lal, C., 2009b. Synthesis, physicochemical propertiesand antimicrobial activity of some new benzimidazole derivatives.Eur. J. Med. Chem. 44, 4028.
5. Bansal, R.K., 2002. Herocyclic Chemistry, third ed. Publisher, New Delhi, New Age International, 401 pp. Bishnoi, A., Pandey, V.K., Saxena, R., 1978. Synthesis and characterization of benzimida-zolylphenothiazine derivatives and a study of their antiviral and antifungal activities. Ind. J. Chem. 41B, 1978.
6. Bistrzycki, Ulffers, 1980. Ueber Diacyl-o-Diamine. Ber. 23, 1876.Campbell, W.C., Denham, D.A., 1983. Chemotherapy. In: Campbell, W.C. (Ed.), Trichinilla and Trichinosis. Plenum Press, USA and London, UK, 340 pp.
7. Demirayak, S., Mohsen, U.A., Karaburun, A.C., 2002. Synthesis andanticancer and anti-HIV testing of some pyrazino[1,2-a]benzimidazole derivatives. Eur. J. Med. Chem. 37, 255. Evans, D., Hicks, T.A., Williamson, W.R.N., Meacock, S.C.R., Kitchen, E.A., 1996. Synthesis of a group of 1H–benzimidazoles and their screening for antiinflammatory activity. Eur. J. Med.Chem. 31, 635.
8. Fischer, Veiel., 1905. The chemistry of benzimidazoles. Ber. 38, 320.Gaba, M., Sing, D., Singh, S., Sharma, V., Gaba, P., 2010. Synthesis and pharmacological evaluation of novel 5-substituted-1-(phenylsulfonyl)-2-methylbenzimidazole derivatives as anti-inflammatory and analgesic agents. Eur. J. Med. Chem. 45, 2245
9. Shaharyar, M., & Mazumder, A. (2017). Benzimidazoles: A biologically active compounds. *Arabian Journal of Chemistry*, *10*, S157-S173.
10. Brauer, V. S., Rezende, C. P., Pessoni, A. M., De Paula, R. G., Rangappa, K. S., Nayaka, S. C., ... & Almeida, F. (2019). Antifungal agents in agriculture: Friends and foes of public health. *Biomolecules*, *9*(10), 521.
11. Tahlan, S., Kumar, S., & Narasimhan, B. (2019). Pharmacological significance of heterocyclic 1 H-benzimidazole scaffolds: a review. *BMC chemistry*, *13*(1), 1-21.
12. Kalarani, R., Sankarganesh, M., Kumar, G. V., & Kalanithi, M. (2020). Synthesis, spectral, DFT calculation, sensor, antimicrobial and DNA binding studies of Co (II), Cu (II) and Zn (II) metal complexes with 2-amino benzimidazole Schiff base. *Journal of Molecular Structure*, *1206*, 127725.
13. Wang, S. Q., Wang, Y. F., & Xu, Z. (2019). Tetrazole hybrids and their antifungal activities. *European journal of medicinal chemistry*, *170*, 225-234.
14. Kalarani, R., Sankarganesh, M., Kumar, G. V., & Kalanithi, M. (2020). Synthesis, spectral, DFT calculation, sensor, antimicrobial and DNA binding studies of Co (II), Cu (II) and Zn (II) metal complexes with 2-amino benzimidazole Schiff base. *Journal of Molecular Structure*, *1206*, 127725.
15. Rashid, Mohammad. "Design, synthesis and ADMET prediction of bis-benzimidazole as anticancer agent." *Bioorganic chemistry* 96 (2020): 103576.
16. Mahida, J., & Patel, R. B. Green Reusable Catalyst For The Formation Diastereo Selective Synthesis Of Cyclohexanones By Condensation Reaction Acetoacetanilide With Different Aromatic Aldehydes At Variety Method Of Reaction.
17. Mahida, J., & Patel, R. B. (2019). Application of Silica Supported Aluminium Hydroxide as a Low Cost Green Catalyst for the One Pot MCR Sythesis via Thermal Method. *Journal of Drug Delivery and Therapeutics*, *9*(4-s), 43-47.
18. Mahida, J., & Patel, R. B. (2019). Application of Silica Supported Aluminium Hydroxide as a Low Cost Green Catalyst for the One Pot MCR Sythesis via Thermal Method. *Journal of Drug Delivery and Therapeutics*, *9*(4-s), 43-47.
19. Mohareb, R. M., Abdallah, A. E. M., & Mohamed, A. A. (2018). Synthesis of novel thiophene, thiazole and coumarin derivatives based on benzimidazole nucleus and their cytotoxicity and toxicity evaluations. *Chemical and Pharmaceutical Bulletin*, *66*(3), 309-318.
20. Brandão, P., Pineiro, M., & Pinho e Melo, T. M. (2019). Flow chemistry: Towards a more sustainable heterocyclic synthesis. *European Journal of Organic Chemistry*, *2019*(43), 7188-7217.
21. Kolarević, A., Ilić, B. S., Anastassova, N., Mavrova, A. T., Yancheva, D., Kocić, G., & Šmelcerović, A. (2018). Benzimidazoles as novel deoxyribonuclease I inhibitors. *Journal of cellular biochemistry*, *119*(11), 8937-8948.
22. Wang, Z., Deng, X., Xiong, S., Xiong, R., Liu, J., Zou, L., ... & Tang, G. (2018). Design, synthesis and biological evaluation of chrysin benzimidazole derivatives as potential anticancer agents. *Natural product research*, *32*(24), 2900-2909.
23. Chaves, S., Resta, S., Rinaldo, F., Costa, M., Josselin, R., Gwizdala, K., ... & Santos, M. A. (2020). Design, synthesis, and in vitro evaluation of hydroxybenzimidazole-donepezil analogues as multitarget-directed ligands for the treatment of Alzheimer's disease. *Molecules*, *25*(4), 985.
24. Cicco, L., Dilauro, G., Perna, F. M., Vitale, P., & Capriati, V. (2021). Advances in deep eutectic solvents and water: applications in metal-and biocatalyzed processes, in the synthesis of APIs, and other biologically active compounds. *Organic & Biomolecular Chemistry*, *19*(12), 2558-2577.
25. Fanjul-Mosteirín, N., & del Amo, V. (2021). Organocatalytic transformations in deep eutectic solvents: Green methodologies made greener. *Tetrahedron*, *84*, 131967.
26. Xie, Q., Zheng, X., Li, L., Ma, L., Zhao, Q., Chang, S., & You, L. (2021). Effect of Curcumin Addition on the

Properties of Biodegradable Pectin/Chitosan Films. *Molecules*, *26*(8), 2152.
27. Delavault, A., Opochenska, O., Laneque, L., Soergel, H., Muhle-Goll, C., Ochsenreither, K., & Syldatk, C. (2021). Lipase-Catalyzed Production of Sorbitol Laurate in a "2-in-1" Deep Eutectic System: Factors Affecting the Synthesis and Scalability. *Molecules*, *26*(9), 2759.
28. Queda, F., Calò, S., Gwizdala, K., Magalhães, J. D., Cardoso, S. M., Chaves, S., ... & Santos, M. A. (2021). Novel Donepezil–Arylsulfonamide Hybrids as Multitarget-Directed Ligands for Potential Treatment of Alzheimer's Disease. *Molecules*, *26*(6), 1658.
29. Hollenbach, R., Ochsenreither, K., & Syldatk, C. (2021). Parameters Influencing Lipase-Catalyzed Glycolipid Synthesis by (Trans-) Esterification Reaction.
30. synthesis & Analysis of some novel amine based heterocyclic compounds and its derivatives prepare by different catalyst concentration and time duration J MAHIDA IJSART 4 (2), 25